

INTERNATIONAL CL. A [illeg.]

GERMAN



PATENT OFFICE

**PATENT APPLICATION PUBLISHED  
FOR OPPOSITION**

**1 012 730**

**W 18920 IVa / 30h**

**APPLICATION DATE:  
ANNOUNCEMENT OF  
THE APPLICATION AND  
PUBLICATION OF THE  
PATENT APPLICATION:**

**24 APRIL 1956**

**25 JULY 1957**

**1**

Of the modern lipstick compounds, it is known that their composition is quite diverse and varied. For example, they contain vegetable oils such as castor oil, sesame oil, olive oil, arachis oil (also in hydration), coconut oil, pumpkin seed oil, almond oil, cocoa butter (also in hydration), and furthermore hydrocarbons or mixtures thereof, such as paraffin, vaseline, vaseline- or paraffin oil, ceresine, ozokerite, highly viscous mineral oil, furthermore waxes from animal and vegetable sources, such as mineral wax, beeswax, as well as fatty alcohols such as stearyl alcohol, cetyl alcohol, furthermore fatty acid esters such as isopropyl myristinate and -palmitate, cetyl ricinoleate, cetyl lauromyristinate, glycerin mono-oleate, polyoxy ethylene mono-laurate and animal fats such as pig fat, beef tallow and other substances such as glycols and substances to increase the solubility of the colorants.

It is known that the composition of a basic lipstick compound and/or the body of the lipstick is an extremely difficult problem. Even with knowledge of the raw materials, extensive experimentation is needed to arrive at a useable recipe. Thus, the aim is to reach the desired objective with as few raw materials as possible. But even today, the recommended mixtures are those of carnauba wax, beeswax, paraffin, ozokerite, castor oil, lanolin, propylene glycol and polyoxy ethylene mono-laurate,

**Means for Coloring Lips, Eyebrows and  
Eyelashes**

**Applicant:  
Wella Aktiengesellschaft  
Darmstadt, Gerauer Allee 65**

**Dr. Hans Freytag, Certified Engineer,  
residing in Fulda,  
was named as the inventor**

aside from other additives, such as colorants. It goes without saying that mastering this type of system is possible only by means of extensive experience. One cannot predict potential processes that might occur during the melt and casting or during storage, and which might render the finished stick compound unusable. In addition, experience has shown that there is also the fact that the raw materials are by no means supplied with consistent quality; slight, barely noticeable or chemically not detectable deviations may pose a challenge to the finished recipe. However, the experts are also of the opinion that the chemical and technical proposals for innovations known so far are not a reason to abandon the body of knowledge in this area; rather, the attempt

should be to provide the lipsticks with skin care properties.

The individual raw materials have various disadvantages. For example, the castor oil is not soluble in mineral oils; therefore, they need to be replaced with animal and vegetable fats and waxes. Although castor oil rarely becomes rancid, it has a tendency toward rancidity in the presence of eosine and thus becomes less compatible to the skin. Other vegetable oils, unless they were hydrated, become rancid easily, and even with hydration, they are not completely tasteless and odorless. Although carnauba wax displays favorable properties, an excessive addition of carnauba wax increases the risk of breakage for the stick compound. Lanoline, on the other hand, easily takes up water, which proves detrimental to the appearance and durability of the layer of color on the lips because the formation of emulsions leads to milky-white color hues. Another disadvantage is that it is difficult to mask the often unpleasant odor. Cetyl alcohol, like oleyl alcohol, lends the sticks a "fatty taste" that is hard to mask. Some of the fatty acid esters, such as butyl stearate, easily sweat out of the compound.

Almost every raw material has these or similar disadvantages, which not only are transferred to the system of the stick compound, but add up there.

It was now discovered that the aforementioned notorious difficulties related to lipstick production can be circumvented in accordance with the invention in an easy manner and the many chemically diverse substances can be reduced to a single group of substances having members that are of optimum chemical relationships if the basic lipstick compounds are composed of silane derivatives, which, as is known, are not part of the silicones.

These silane derivatives refer to tetraalkoxy silanes, alkyl trialkoxy silanes, aryl trialkoxy silanes, dialkyl dialkoxy silanes, diaaryl dialkoxy silanes, and alkyl aryl dialkoxy silanes. In this context, the term alkyl refers to saturated and unsaturated hydrocarbon groups with 1 to 20 carbon atoms, the term aryl, for example, refers to

phenyl-, benzyl-,  $\alpha$ - or  $\beta$ -naphthyl groups. The alkyls (alkoxy groups) linked to the silicon atom by an oxygen atom, on the other hand, are supposed to represent hydrocarbon groups of 8 to 30 saturated or unsaturated carbon atoms. The various alkoxy silanes described in detail above are materials that are easily available technically. They are obtained by re-estriification of appropriate ethoxy silanes with the aforementioned fatty alcohols, either by dissolving the reaction partners in an equimolar quantity or the fatty alcohols in a sufficient excess of the respective ethoxy silane and heating the solution to 180 °C while introducing dry hydrogen chloride, with the ethyl alcohol escaping. Re-estriification can also be achieved with the use of zinc dust. In that case, heating without hydrogen chloride units is sufficient. Sometimes, this type of re-estriification is even preferable. It goes without saying that silanes can be produced with similar success by reacting the appropriate halogen silanes with the fatty alcohols (see B. Helferich and J. Hansen, Reports by the German Chemical Association, 57, page 795 [1924]). The reaction liquid is mixed with ether in which the excess ethoxy silanes and the produced higher alkoxy silane are soluble, and is precipitated with alcohol (the means for dissolution and re-precipitation may be varied depending on the requirements and the solubility behavior of the finished silane). The re-precipitation is repeated two-to four times, depending on the required purity, and the last rests of the dissolution-and precipitation means on the water bath are removed by heating in vacuum. The use of methoxy silanes is risky because this compound is extremely toxic in vapor form.

Unlike the silicates of the lower molecular alcohols, the alkoxy silanes used for the production of the lipstick compound in accordance with the invention are largely resistant compounds that are completely stable to water, whereas tetra ethoxy silane, for example, is sensitive to water. Furthermore, the alkoxy silanes used in accordance with the invention are physiologically safe as long as they are pure,

i.e., as long as they do not contain any traces of hydrochloric acid or of solvents from their production. It should be noted that already some time ago, esters of the orthosilicic acid were detected in organs and in the blood, which therefore may be of significance for the silicon metabolism.

Depending on the substituents, the compounds characterized above represent substances that are oily, pasty, and already melt at body heat, or hard, higher-melting colorless substances, with a slightly oily or buttery and sometimes flowery taste and an unobtrusive odor. All compounds can be mixed smoothly in liquid condition. Mixtures of practically any consistency can be produced with appropriate parts of usually two to four of said alkoxy silanes. They easily dissolve fat dyes, and the generally known pigments for lipsticks are just as easily dispersible in the melts. Because of the very scant self-smell, the perfuming does not create any difficulties whatsoever, and neither does the addition of aromas. Unlike with the previously used raw materials, the compounds are selected from within the most closely related, precisely defined compositions of reproducible purity. By knowing the correlation between constitution and properties, the use of the few components can be predetermined with a large degree of certainty.

In accordance with the invention, it is possible to produce cosmetic compounds in general, such as eyebrow sticks and lash sticks. The means in accordance with the invention can be brought into liquid, semi-solid or solid form and filled into bottles, tubes, and the like depending, on the consistency. In addition to fat dyes, which have an excellent solubility in the alkoxy silanes, the common coloring substances, such as pigment coloring substances and eosine acids, can be dispersed very finely. Other random active substances can also be integrated easily into the compounds.

#### Example 1

66.7 parts-by-weight phenyl tricetoxy silane [ $C_6H_5Si(OC_{16}H_{33})_3$ ] (also referred to

as phenyl tricetylorthosilicate), white compound with a melting point of 36 to 38 °Celsius, is mixed with 11.1 parts-by-weight tetraoleoxysilane [ $Si(OC_{18}H_{35})_4$ ] (also referred to as tetraoleylorthosilicate), a pale yellowish oily liquid, and 11.1 parts-by-weight tetrastearoxysilane [ $(C_{18}H_{37}O)_4Si$ ], are melted thoroughly at approximately 60 to 70 °Celsius, and 11.1 parts-by-weight permanent bordo FRF extra powder are added, which is 2-methyl-4-nitro-aminobenzol → 1-(2', 3' - oxynaphthoylamino); 2-methylbenzol, listed in the List of Pigments and Coloring Substances for Cosmetics of the Deutsche Forschungsgemeinschaft [German Research Group], Commission for the Work on the Foodstuff Coloring Substance Problem, Communication No. 3 (1952), page 10.

The compound is poured into molds and the result is a strongly glistening, very soft, well covering stick.

#### Example 2

44.25 parts-by-weight phenyl-tricetoxy-silane, 8.85 parts-by-weight phenyl-trioleoxysilane (also referred to as phenyltrioleylorthosilicate), yellowish, oily liquid, 8.85 parts-by-weight tetrastearoxysilane and 26.55 parts-by-weight of a mixture of tetracetoxysilane and tetra-stearoxysilane, obtained with the re-estification of a commercial mixture of cetyl- and stearyl alcohol, are melted as in Example 1 and mixed with 10.6 parts-by-weight permanent carmine FB extra powder by Farbwerke Hoechst, which is 2-methoxy-5-diethylsulfamino-1-aminobenzol → 1-(2', 3' - oxynaphthoylamino) -2, 4-dimethoxy-5-chlorbenzol, listed in the List of Pigments and Coloring Substances for Cosmetics cited above, and then 0.9 parts-by-weight of a perfume oil are added.

The compound is poured in molds and the result is an excellent lipstick with a dropping point at 48 to 49 °Celsius.

### Example 3

57.7 parts-by-weight phenyltricetylorthosilicate, 38.5 parts-by-weight tetrastearylorthosilicate, 3.8 parts-by-weight permanent carmine FB extra powder.

The result is a lipstick with a dropping point of 45 °Celsius.

### Example 4

35.0 parts-by-weight penyltrimyristylorthosilicate, 65 parts-by-weight tetraarachylorthosilicate is melted in the water bath at 75 °Celsius and mixed, and 0.005 parts-by-weight vitamin A acetate (commercially known under the patented title Arovit) or 0.007 parts-by-weight vitamin A stearate (commercially known under the patented title Adaptilol) are added and mixed with 10.6 parts-by-weight permanent carmine FB extra powder.

The lipstick obtained in this manner has a dropping point of 60 °Celsius.

### Example 5

When using pure carbon instead of the red pigment colorings in the preceding examples, the result are black covering eyebrow- and eyelash sticks.

By determining the appropriate quantity ratios, it is possible to produce sticks of any appearance, any hardness, and practical constancy from only a few alkoxy silanes, such as tetradecyl-, phenyltridecyl-, tetralauryl-, phenyltrilauryl-, tetramyristyl-, phenyltrimyristyl, tetracetyl-, phenyltricetyl-, tetrastearyl-, phenyltristearyl- as well as tetraoleyl- and phenyltrioleylorthosilicates. The phenyl group at the Si atom exercises a softening effect.

### PATENT CLAIMS

1. Means for coloring lips, eyebrows and eyelashes, characterized in that the basic compound is comprised of mixtures of tetraalkoxy silanes and/or alkyl trialkoxy silanes and/or aryltralkoxy silanes and/or alkyl-aryl dialkoxy silanes and/or dialkyl dialkoxy silanes and/or diaryl dialkoxy silanes.

2. Means in accordance with Claim 1, characterized in that the alkoxy groups correspond to the formula  $-\text{O C}_n\text{H}_{2n-1}$ , with  $n$  having the value 8 to 30.

3. Means in accordance with the claims 1 and 2, characterized in that the radicals linked directly to the silicon atom correspond to the formula  $-\text{C}_n\text{H}_{2n+1}$ ,  $-\text{C}_n\text{H}_{2n-1}$  and  $-\text{C}_n\text{H}_{2n-3}$ , with  $n$  being equal to 1 to 20 and/or corresponding to the phenyl-,  $\alpha$ - or  $\beta$ -naphthyl- or benzyl group.

4. Means in accordance with the claims 1 to 3, characterized in that it comprises fat soluble coloring substances and vitamins.